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(6)

QUALIFICATION OF A NEW MAPO SOURCE AND ERL-510 CURING AGENT FOR MINUTEMAN STAGE 1 UF-2121 LINER.

Technical report by John A. Thompson

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ABSTRACT

Thiokol Chemical Corporation/Wasatch Division uses MAPO in the production of UF-2121 liner. Thiokol changed MAPO vendors and, therefore, qualification of the new source MAPO was required.

Thiokol prepared specimens from the new source and also specimens from the original source which are to be used as the control material in the 10 year surveillance testing program. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.

This report includes the test results for the first, second, third, fourth, fifth, and sixth time testing of the control and special specimens at Ogden ALC.

Statistical analysis of the test data showed that the physical properties of the new source compared closely to the old source of UF-2121 liner specimens.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 REV A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.

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INTRODUCTION

A. PURPOSE:

Quality assurance testing of specimens prepared from the new source of MAPO to assure that liner material for First Stage Minuteman Motors will perform as predicted.

B. BACKGROUND:

*Tris [1-(2 methyl) aziridinyl] phosphine oxide (MAPO) is used as a curing agent in the Minuteman Stage One UF-2121 liner. MAPO was produced by Immont Chemical (Immont) and shipped to Arsynco Incorporated (Arsynco) for purification and marketing. Immont sold the production rights for MAPO to Arsynco and terminated the production of raw MAPO in 1972.

Since MAPO is a critical ingredient in UF-2121 liner formulation, the source change for the manufacture of MAPO was considered a serious change. Therefore, it was necessary for Thiokol to conduct qualification testing on liner material using MAPO manufactured by Arsynco before it could be considered acceptable for use in Minuteman Stage I UF-2121 liner.

ERLA-500 was the qualified epoxy curing agent used with MAPO in the UF2121 liner. Union Carbide terminated their process for ERLA-500. ERLA-510 used in similar liners (i.e. UF-2137) was substituted for ERLA-500 and qualified with MAPO from the new source.

The test conditions and test methods are shown in Table I.

*TWR-7857 Rev A Report, J. W. Rabern

Qualification testing was performed by Thiokol and reported in TWR-7857 Rev A. In addition, specimens were prepared by Thiokol from the new vendor's material and from the old source material for a "follow on" test program. These specimens were then transferred to Ogden ALC for a continuing surveillance test program designed to cover a ten year span. The material from the old source will be used as the control samples.

The ten year sampling plan is shown below. Those specimens identified for the sixth year were tested at this test period. The types of specimens are Disc (steel/liner/steel), Cup (steel/liner/TP-H1011), and Peel (broadcloth/liner/TP-H1011). For the disc specimen, the adhesion between the liner and steel is the critical factor. For the cup specimen, the adhesion between the propellant and the liner is critical. For the peel specimen the propellant to liner peel strength when pulled at 180° is critical.

TEN YEAR CONTINGENCY AGING SAMPLE CODING

Storage Age (yr)	Storage Temp (°F)	Disc (Sample Nr) Control*	Disc (Sample Nr) Special**	Cup (Sample Nr) Control*	Cup (Sample Nr) Special**	Peel (Sample Nr) Control*	Peel (Sample Nr) Special**
1	75	1 - 6	181-186	61-66	241-246	121-126	301-306
2	75	7 -12	187-192	67-72	247-252	127-132	307-312
3	75	13-18	193-198	73-78	253-258	133-138	313-318
4	75	19-24	199-204	79-84	259-264	239-144	319-324
5	75	25-30	205-210	85-90	265-270	145-150	325-330
6	75	31-36	211-216	91-96	271-276	151-156	331-336
7	75	37-42	217-222	97-102	277-282	157-162	337-342
8	75	43-48	223-228	103-108	283-288	163-168	343-348
9	75	49-54	229-234	109-114	289-294	169-174	349-354
10	75	55-60	235-240	115-120	295-300	175-180	355-360

* Liner mix A73-11846 - control or old MAPO source material

** Liner mix A73-11810 - Experimental MAPO

TABLE I

Test Conditions and Methods

Group	Test	Condition	Configuration	G085 Spec Code	Spec Per Cond	Total Number of Spec	Test Method
Bond in Tension Disc	Tensile Adhesion OI#127-3	CHS 0.5 in/min, Chart 5.0 in/min, 500 lbs full scale load 77°F ± 2°	Discs	TV	Control 6 Special 6	12	A
Bond in Tension Cup	Tensile Adhesion OI#127-3	CHS 0.5 in/min, Chart 5.0 in/min, 200 lbs full scale load 77°F ± 2°	Cup	TC	Control 6 Special 6	12	A
180° Peel Specimens	Tensile Peel OI#127-3	CHS 10 in/min 77°F ± 2° Chart 5 in/min 20 lbs full scale load	Peel	TE	Control 6 Special 6	12	B

TEST CONDITIONS

A. Testing of tensile adhesion specimens was performed using an Instron testing instrument. Properties measured were maximum stress to the nearest pound and failure mode.

Steel disc specimens require a stress of about 240 psi. The recommended initial full scale load is 500 pounds. This instrument setting should be changed to another setting if the first reading goes off scale on the high side. If 500 psi is exceeded, then change the reading to 1000 psi full scale.

Cup adhesion specimens are tested with a stress of 200 lbs per sq inch; the recommended full scale load is 500 lbs.

B. Testing of 180° peel samples was performed using an Instron testing instrument. The physical property of the material to be determined was the average peel strength to the nearest pound per inch.

NOTE: Thiokol's procedure for Testing and Laboratory Mixing of UF-2121 Liner. SLP 400, 28 April 71.

STATISTICAL ANALYSIS

UF-2121 liner material is being tested under a ten year program to determine whether or not differences exist between liner materials manufactured from two separate sources of curing agent (MAPO). Test specimens were manufactured in two groups; control, using original source curing agent, and special, using new source curing agent. The test specimens for these two groups are of three kinds; disc, cup, and peel. For each specimen type within each test group the sample test size is six. Laboratory testing for six test periods or six years has been accomplished. Test data for the years 1975, 1976, 1977, 1978, 1979, and 1980 are in Tables 2 through 7, and columns are summarized using means and standard deviations.

With six test periods accomplished, regression plots (Figures 1 - 6) were made to determine whether slope and elevation differences existed between control and special test data. No differences in either slope or elevation were found. The regression model $Y = a + bx$, using individual data points, was used in the regression analyses. The variance about the least squares trend line is used to compute a tolerance interval such that at the 90% confidence level 90% of the sample distribution falls within this interval. This tolerance interval is extrapolated 24 months past the age point pertaining to the oldest specimens tested. The statistical significance of the slope of the trend line is evaluated for each regression plot. If significant, it is an indication that change over time is occuring.

In determining differences in data pertaining to the two MAPO sources, analysis of covariance was employed to compare control and special data from

the regressions for each of the three types of test specimens. For analysis of covariance results, see Table 8. Taken at the five percent significance level, the only difference found was in disc data in the variance of the data away from the regression line.

TEST RESULTS

The 1980 test data and the mean for the respective control and special data are shown in Table 7. In addition, for a convenient comparison, the 1975 through 1979 test data are included in Tables 2 through 6.

The statistical analysis results for the 1980 testing are shown in Table 8 with the regressions shown in Figures 1 through 6.

DISC:

A statistically significant difference is shown for variance of test data in the MAPO source (Table 8) with no significant difference for the slope and elevation of the regression curves (Table 8).

The regression curves show a statistically significant gradual decrease for both the control and special sample data (Figures 1 & 2).

For the year 1980, the mean of the control and special data is 15.06 and 14.28 kg/sq cm respectively.

The mimimum specification requirement according to TWR-7857 REV A, is 12.30 kg/sq cm mimimum. As seen in Table 7, MAKPH's data is well above this mimimum.

The failure mode was 100% cohesive in the liner for both the control and special specimens.

CUP:

There is no significant difference in variance, slope or elevation when comparing control and special regression data (Table 8).

The regression curves show a statistically significant gradual decrease in maximum stress as the specimens age (Figures 3 & 4).

According to TWR-7857 REV A report, the minimum requirement is 4.92 kg/sq cm. The data means are 10.00 kg/sq cm for the control and 10.49 kg/sq cm for the special specimens.

The failure mode for the control and special specimens was 100% adhesive liner to propellant.

PEEL:

No significant difference is shown for the variance, slope or elevation when comparing control with special regression data (Table 8).

The regression curves (Figures 5 & 6) show a statistically significant gradual increase in peel strength with respect to the age of the specimens.

Thiokol reported (TWR-7857 REV A) 0.679 and 0.732 *kg/L cm respectively for the control and special mean data at age six months. This compares with 1980 data of 1.09 and 1.08 kg/1 cm respectively for control and special mean data (Table 7).

The mode of failure was 100% liner to propellant bond.

* Kilograms per linear centimeter

CONCLUSIONS

Based on this analysis, the only statistically significant difference between the control and special specimens is the variance for disc specimens.

The regressions show a gradual statistically significant change.

The strength of specimens is well above the required minimum for disc and cup, and above that reported in Thiokol testing for peel.

From the data analysis, the new source of raw material performs as well as the old source; and therefore is expected to perform satisfactorily.

RECOMMENDATIONS

It is recommended that the testing plan be continued to assure long range capability of the liner produced from the new source of material.

TABLE 2

TEST DATA SUMMARY
JULY 1975

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/L-Cm	Nr	Kg/L-Cm
1	17.085	181	17.225	61		241	11.601	121	0.6786	301	0.6786
2	17.507	182	17.858	62	11.812	242	11.601	122	0.6786	302	0.6965
3	17.225	183	17.015	63	11.741	243	11.531	123	0.7143	303	0.6965
4	17.929	184	16.944	64	12.163	244	11.671	124	0.7500	304	0.6965
5	17.366	185	17.436	65	12.234	245	11.390	125	0.7679	305	0.7143
6	17.296	186	19.054	66	11.882	246	11.390	126	0.7858	306	0.6965
$\bar{Y}$	17.401		17.589		11.966		11.531		0.7292		0.6965
S	0.2943		0.7899		0.2191		0.1176		0.0458		0.0113

TABLE 3

TEST DATA SUMMARY
MAY 1976

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/L-Cm	Nr	Kg/L-Cm
7	15.116	187	14.483	67		247	13.288	127	0.9643	307	1.0536
8	16.311	188	14.905	68	13.710	248	13.710	128	0.9286	308	1.0358
9	15.397	189	14.483	69	13.640	249	13.640	129	0.9286	309	1.0179
10	15.960	190	14.765	70	13.007	250	13.077	130	1.0179	310	1.0358
11	15.819	191	15.468	71	13.148	251	13.359	131	1.1072	311	1.0536
12	14.554	192	14.765	72	13.499	252	13.499	132	1.0001	312	1.0358
$\bar{Y}$	15.526		14.812		13.401		13.429		0.9911		1.0388
S	0.6356		0.3633		0.3088		0.2354		0.0675		0.0134

NOTE: Kg/L-Cm = Kilograms per linear centimeter. Also, for the peel test the results are in average peel.

TABLE 4

TEST DATA SUMMARY
APRIL 1977

	DISC				CUP				PEEL			
	Control		Special		Control		Special		Control		Special	
Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/L-Cm	Nr	Kg/L-Cm
13	17.155	193	17.436	73	9.281	253	9.140	133	0.7522	313	0.7858
14	16.522	194	16.874	74	9.281	254	9.070	134	0.7143	314	0.7858
15	16.874	195	16.944	75	8.999	255	8.999	135	0.6786	315	0.7858
16	17.015	196	17.366	76	9.281	256	9.140	136	0.7500	316	0.7143
17	16.874	197	17.015	77	9.492	257	8.367	137	0.7500	317	0.7500
18	16.874	198	17.015	78	9.281	258	8.789	138	0.6429	318	0.7143
$\bar{X}$	16.886		17.108		9.269		8.918		0.7113		0.7560
S	0.2107		0.2337		0.1570		0.2994		0.0429		0.0352

TABLE 5

TEST DATA SUMMARY
JUNE 1978

	DISC				CUP				PEEL			
	Control		Special		Control		Special		Control		Special	
Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/Cm^2	Nr	Kg/L-Cm	Nr	Kg/L-Cm
19	14.906	199	14.554	79	10.898	259	10.406	139	0.9109	319	1.0180
20	14.624	200	14.695	80	10.968	260	10.476	140	0.9823	320	1.0359
21	14.695	201	14.343	81	10.617	261	10.616	141	0.9466	321	1.0180
22	14.906	202	14.343	82	10.125	262	10.125	142	1.0002	322	1.0359
23	14.343	203	14.624	83	10.406	263	10.687	143	1.0716	324	1.0537
24	15.187	204	14.414	84	10.476	264	10.125	144	0.9466	326	0.9287
$\bar{X}$	14.777		14.496		10.582		10.406		0.9764		1.0150
S	0.2898		0.1503		0.3166		0.2391		0.0561		0.0444

TABLE 6

TEST DATA SUMMARY
11 JUNE 1979

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	kg/cm^2	Nr	kg/cm^2	Nr	kg/cm^2	Nr	kg/cm^2	Nr	kg/L-cm	Nr	kg/L-cm
25	17.015	205	17.436	85	9.632	265	9.562	145	0.8572	325	0.7679
26	16.874	206	16.874	86	9.703	266	9.140	146	0.8393	326	0.7679
27	16.874	207	16.593	87	9.773	267	9.562	147	0.8572	327	0.9643
28	16.944	208	16.522	88	9.632	268	9.281	148	0.8572	328	0.7143
29	16.804	209	16.382	89	9.632	269	9.421	149	0.7679	329	0.7143
30	16.171	210	17.366	90	9.492	270	9.421	150	0.8036	330	0.7500
$\bar{Y}$	16.780		16.862		9.644		9.398		0.8304		0.7798
S	0.3070		0.4477		0.0934		0.1644		0.0370		0.0936

TABLE 7

TEST DATA SUMMARY
SEPTEMBER 1980

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	kg/cm^2	Nr	kg/cm^2	Nr	kg/cm^2	Nr	kg/cm^2	Nr	kg/L-cm	Nr	kg/L-cm
31	15.116	211	14.764	91	9.281	271	11.109	151	0.9465	331	1.1965
32	14.905	212	14.413	92	10.054	272	10.265	152	1.0179	332	1.1786
33	14.624	213	14.202	93	10.616	273	10.898	153	1.0715	333	1.1072
34	14.905	214	14.272	94		274	10.687	154	1.1072	334	1.0536
35	15.678	215	13.870	95	9.894	275	9.491	155	1.0179	335	1.0358
36	15.116	216	14.272	96	10.054	276		156	1.0715	336	1.0358
$\bar{Y}$	15.057		14.284		9.998		10.490		1.0388		1.0846
S	0.3539		0.3188		0.4750		0.6397		0.0569		0.0613

TABLE 8.

ANALYSIS OF COVARIANCE RESULTS WHEN COMPARING

CONTROL AND SPECIAL REGRESSION DATA

Type of Data Compared	Parameter Compared	F-value	Degrees of Freedom	Significance
Disc	Variance	1.83	34, 34	significant
	Slope	0.32	1, 68	not significant
	Elevation	0.64	1, 69	not significant
Cup	Variance	1.38	33, 31	not significant
	Slope	0.20	1,64	not significant
	Elevation	0.06	1, 65	not significant
Peel	Variance	1.49	34, 34	not significant
	Slope	0.06	1, 68	not significant
	Elevation	0.32	1. 69	not significant

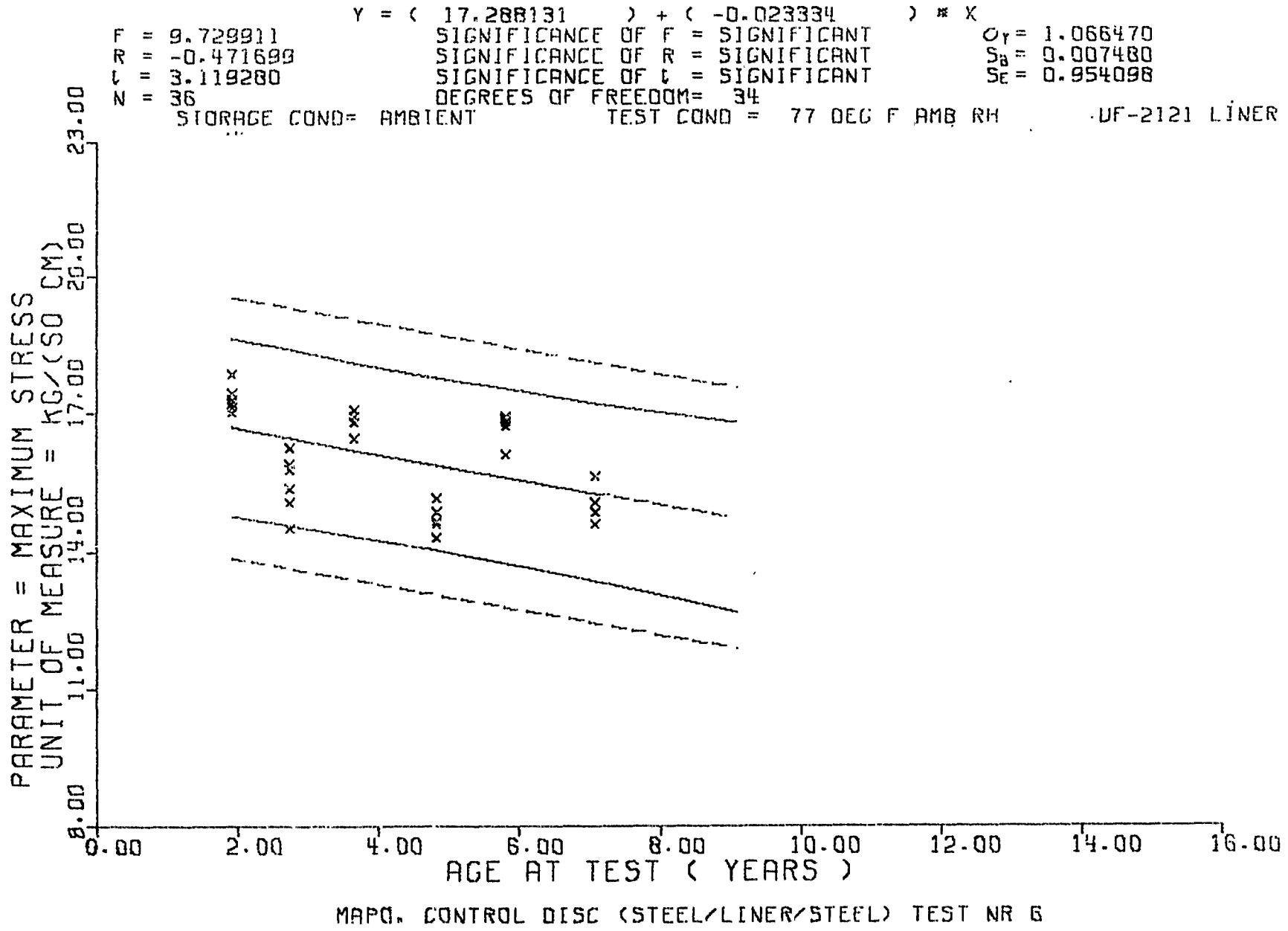
Y = (17.288131) + (-0.023334) * X
F = 9.729911
R = -0.471699
t = 3.119280
N = 36
SIGNIFICANCE OF F = SIGNIFICANT
SIGNIFICANCE OF R = SIGNIFICANT
SIGNIFICANCE OF t = SIGNIFICANT
DEGREES OF FREEDOM= 34
σY = 1.066470
Sa = 0.007480
Se = 0.954098
STORAGE COND= AMBIENT
TEST COND = 77 DEG F AMB RH
UF-2121 LINER
PARAMETER = MAXIMUM STRESS
UNIT OF MEASURE = KG/(SQ CM)
8.00
11.00
14.00
17.00
20.00
23.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
AGE AT TEST (YEARS)
MAPC. CONTROL DISC (STEEL/LINER/STEEL) TEST NR 6

Figure 1

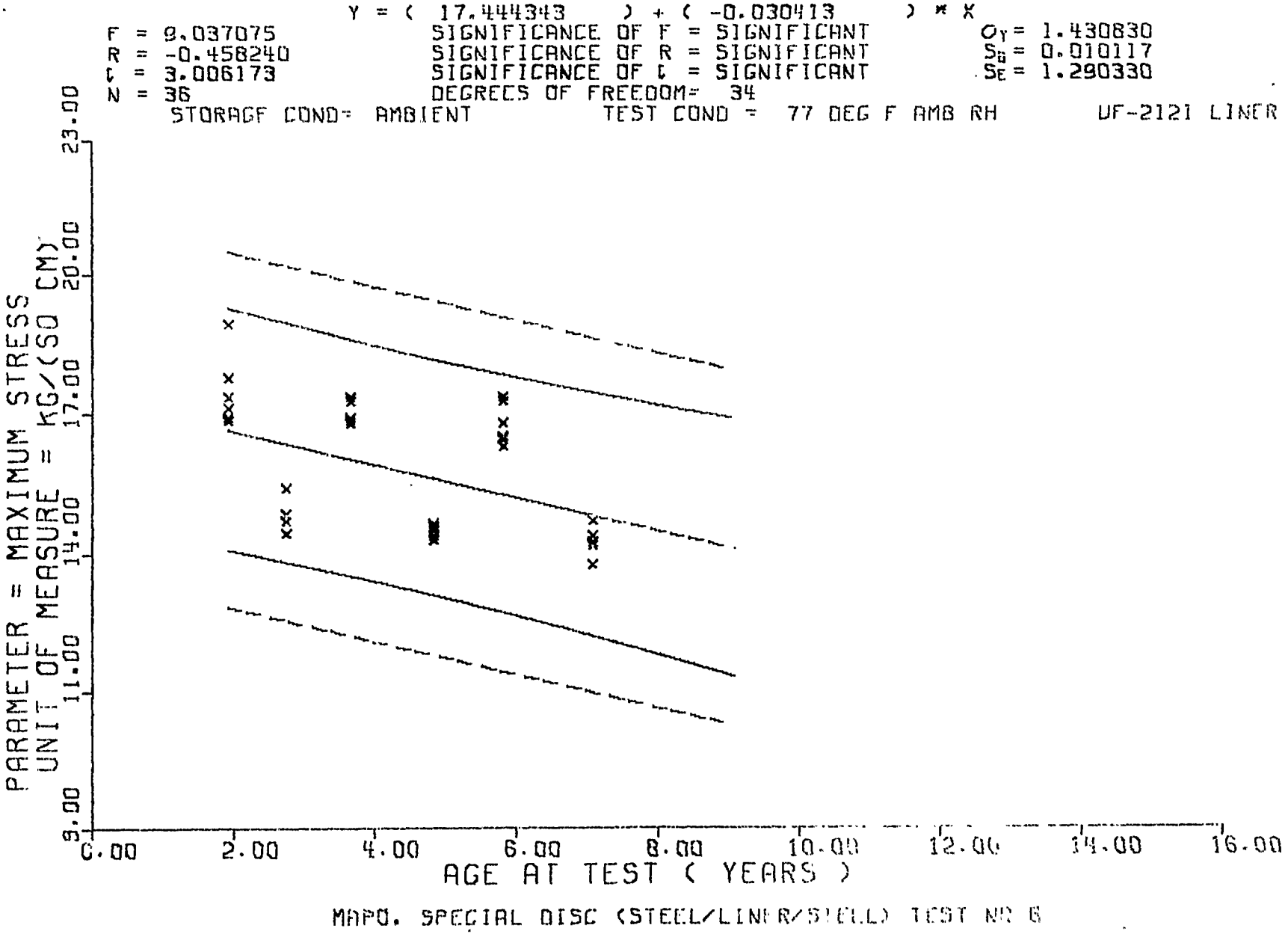
Y = (17.444343) + (-0.030413) * X
F = 9.037075
R = -0.458240
t = 3.006173
N = 36
SIGNIFICANCE OF F = SIGNIFICANT
SIGNIFICANCE OF R = SIGNIFICANT
SIGNIFICANCE OF t = SIGNIFICANT
DEGREES OF FREEDOM= 34
σY = 1.430830
Sb = 0.010117
SE = 1.290330
STORAGE COND= AMBIENT
TEST COND = 77 DEG F AMB RH
UF-2121 LINER
PARAMETER = MAXIMUM STRESS
UNIT OF MEASURE = KG/(SQ CM)
23.00
20.00
17.00
14.00
11.00
8.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
AGE AT TEST (YEARS)
MAPD. SPECIAL DISC (STEEL/LINER/STEEL) TEST NO 6

Figure 2

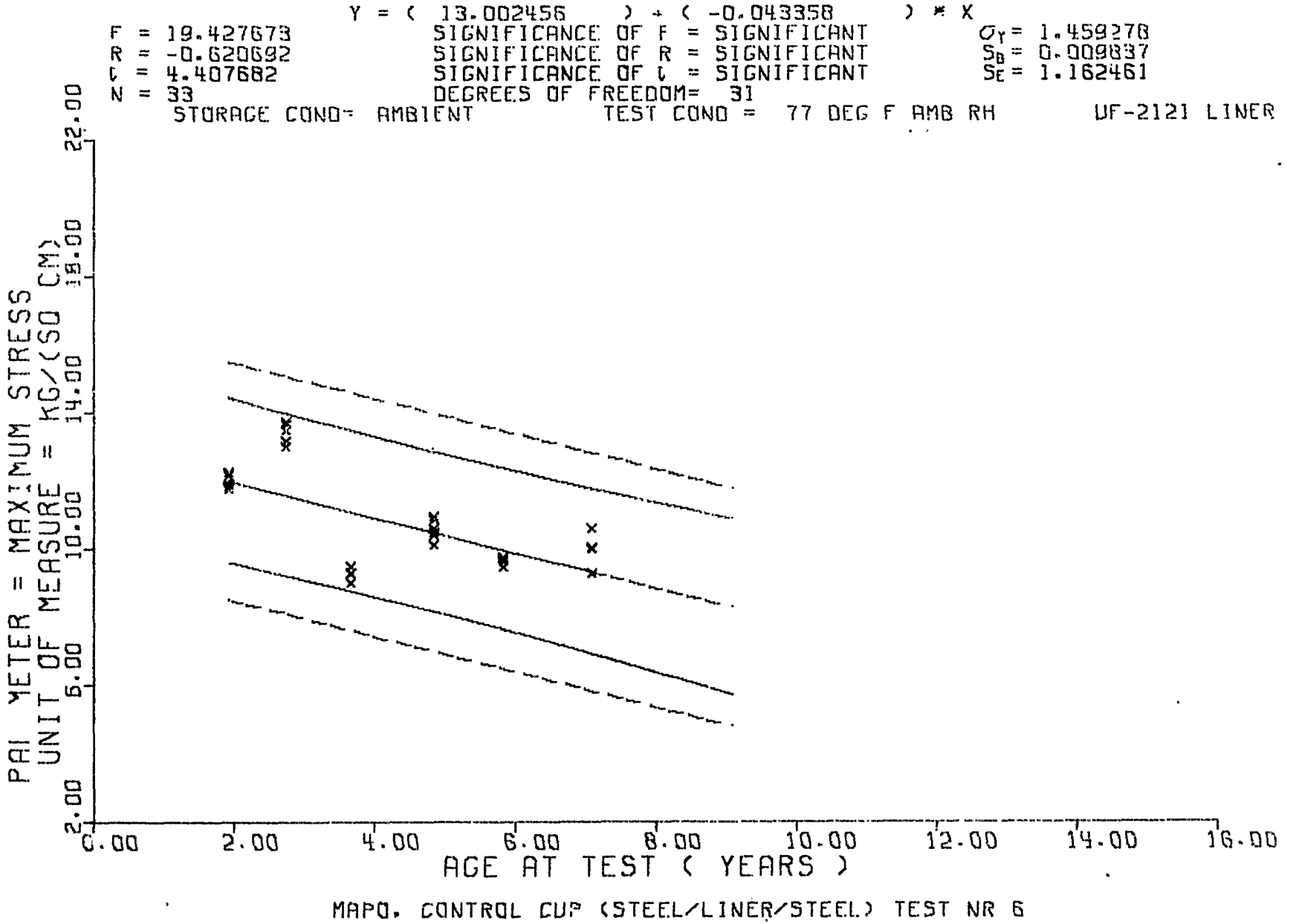
Y = (13.002456) + (-0.043358) * X
F = 19.427673
R = -0.620692
t = 4.407682
N = 33
SIGNIFICANCE OF F = SIGNIFICANT
SIGNIFICANCE OF R = SIGNIFICANT
SIGNIFICANCE OF t = SIGNIFICANT
DEGREES OF FREEDOM= 31
σY = 1.459278
SB = 0.009837
SE = 1.162461
STORAGE COND= AMBIENT
TEST COND = 77 DEG F AMB RH
UF-2121 LINER
PARAMETER = MAXIMUM STRESS
UNIT OF MEASURE = KG/(SQ CM)
22.00
18.00
14.00
10.00
6.00
2.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
AGE AT TEST (YEARS)
MAPO. CONTROL CUP (STEEL/LINER/STEEL) TEST NR 6

Figure 3

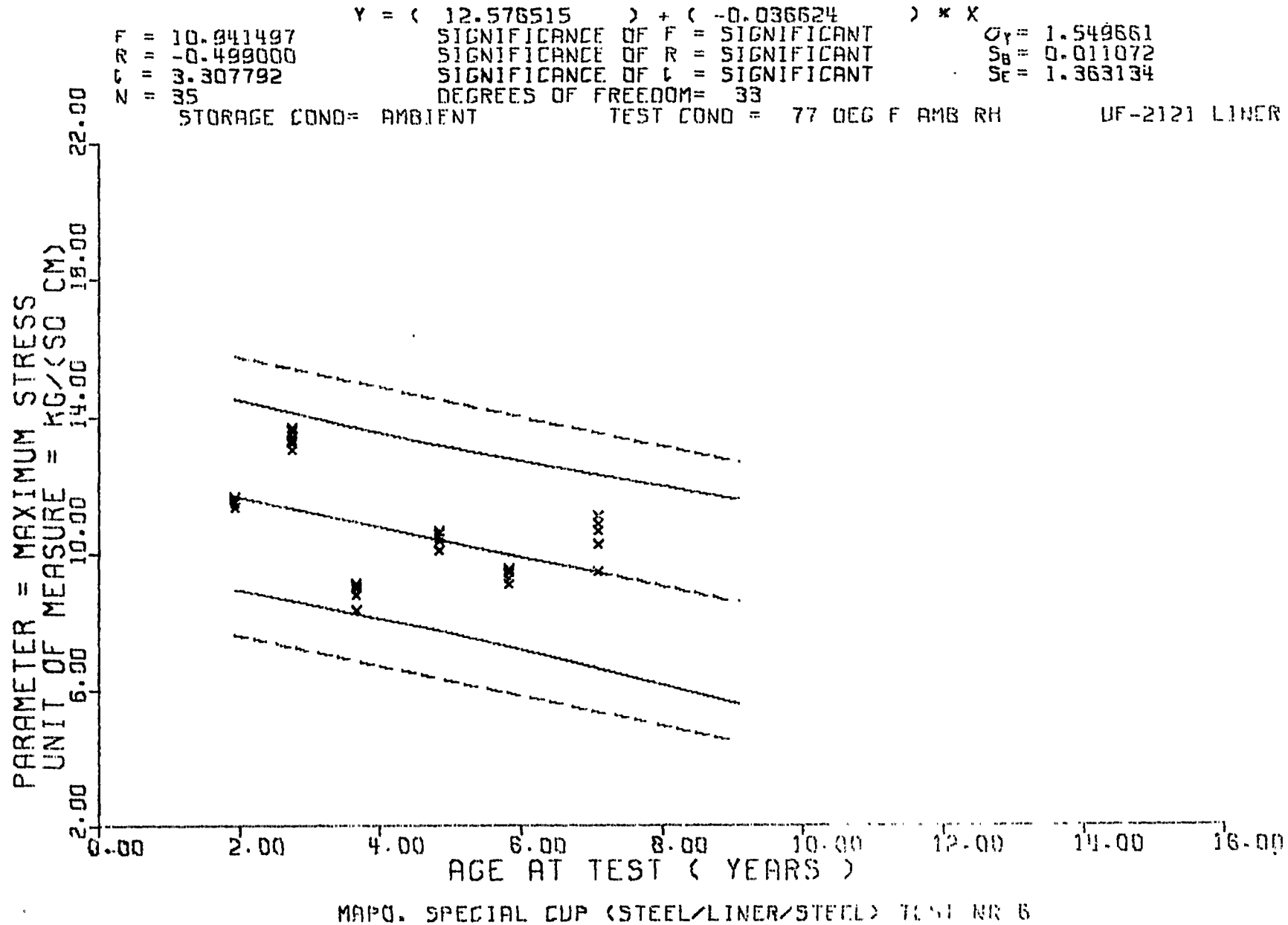
Y = (12.576515) + (-0.036624) * X
F = 10.941497
R = -0.499000
t = 3.307792
N = 35
SIGNIFICANCE OF F = SIGNIFICANT
SIGNIFICANCE OF R = SIGNIFICANT
SIGNIFICANCE OF t = SIGNIFICANT
DEGREES OF FREEDOM= 33
σY = 1.549661
SB = 0.011072
Sε = 1.363134
STORAGE COND= AMBIENT
TEST COND = 77 DEG F AMB RH
UF-2121 LINER
PARAMETER = MAXIMUM STRESS
UNIT OF MEASURE = KG/(SQ CM)
22.00
18.00
14.00
10.00
6.00
2.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
AGE AT TEST (YEARS)
MAPO. SPECIAL CUP (STEEL/LINER/STEEL) TEST NR 6

Figure 4

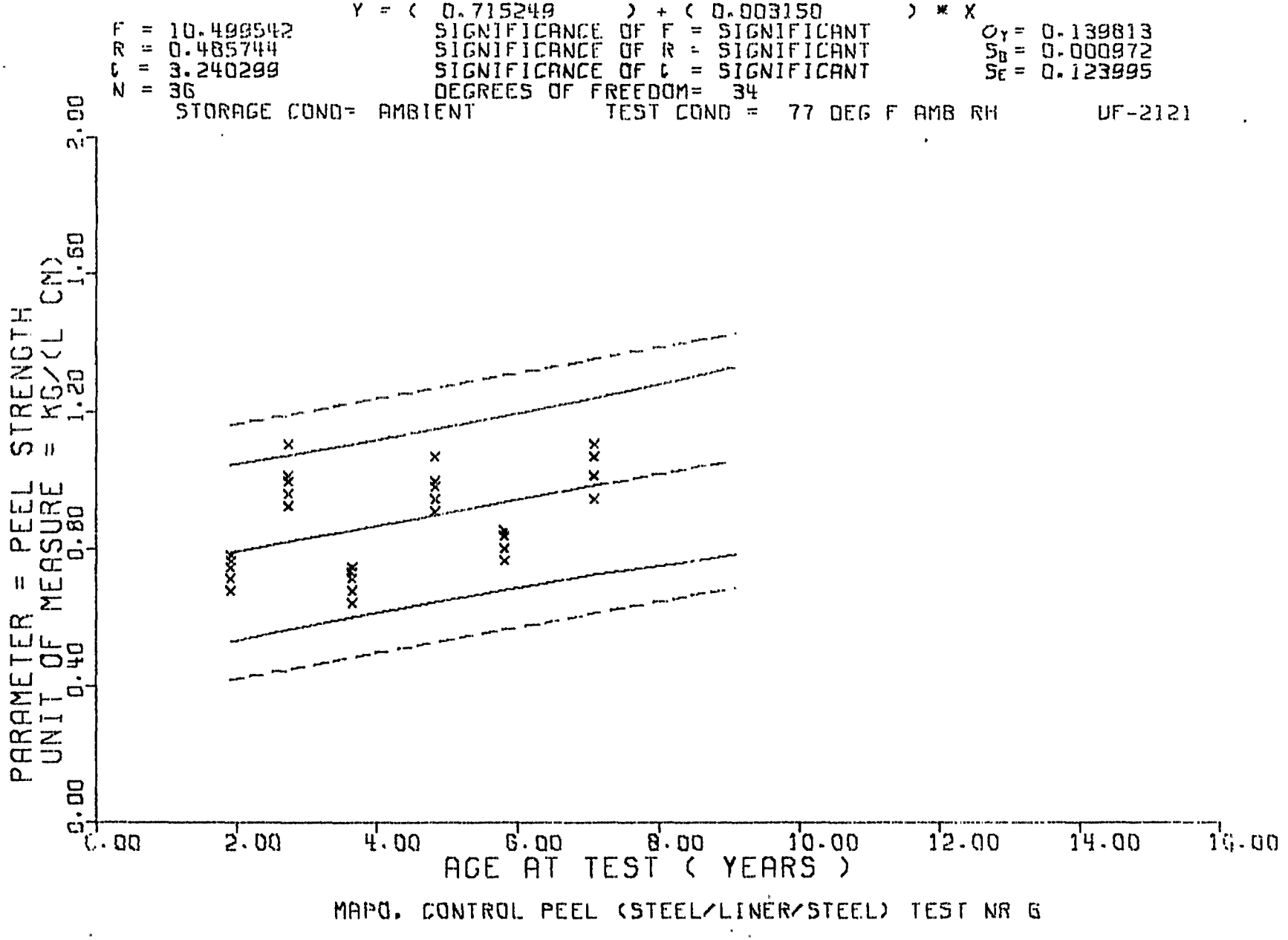
Y = (0.715249) + (0.003150) * X
F = 10.499542
R = 0.485744
t = 3.240299
N = 36
SIGNIFICANCE OF F = SIGNIFICANT
SIGNIFICANCE OF R = SIGNIFICANT
SIGNIFICANCE OF t = SIGNIFICANT
DEGREES OF FREEDOM= 34
σ_Y = 0.139813
S_B = 0.000972
S_ε = 0.123995
STORAGE COND= AMBIENT
TEST COND = 77 DEG F AMB RH
UF-2121
PARAMETER = PEEL STRENGTH
UNIT OF MEASURE = KG/(L CM)
2.00
1.60
1.20
0.80
0.40
0.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
AGE AT TEST (YEARS)
MAPO. CONTROL PEEL (STEEL/LINER/STEEL) TEST NR 6

Figure 5

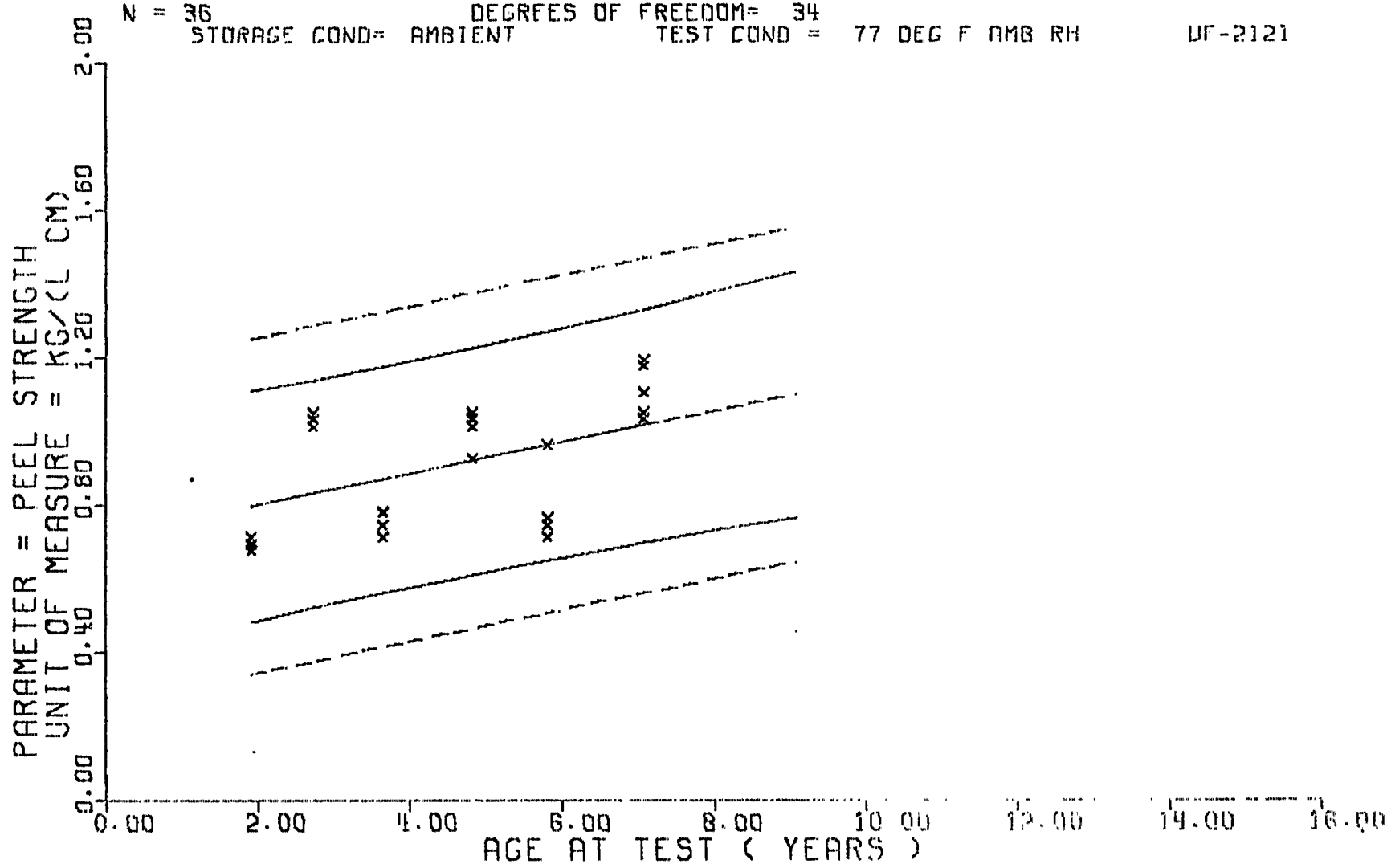
Y = (0.713247) + (0.003540) * X
F = 8.879981 SIGNIFICANCE OF F = SIGNIFICANT
R = 0.455070 SIGNIFICANCE OF R = SIGNIFICANT
t = 2.979929 SIGNIFICANCE OF t = SIGNIFICANT
N = 36 DEGREES OF FREEDOM= 34
σT = 0.167730
SB = 0.001188
SE = 0.151537
STORAGE COND= AMBIENT TEST COND = 77 DEG F AMB RH UF-2121
PARAMETER = PEEL STRENGTH
UNIT OF MEASURE = KG/(L CM)
0.00
0.40
0.80
1.20
1.60
2.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
AGE AT TEST (YEARS)
MAPD. SPECIAL PEEL (STEEL/LINER/STEEL) TEST NR 6

Figure 6

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Minuteman
MAPO
UF-2121 Liner

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Thiokol Chemical Corporation/Wasatch Division uses MAPO in the production of UF-2121 liner. Thiokol changed MAPO vendors and, therefore, qualification of the new source MAPO was required.

Thiokol prepared specimens from the new source and also specimens from the original source which are to be used as the control material in the 10 year surveillance testing program. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.

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This report includes the test results for the first, second, third, fourth, fifth, and sixth time testing of the control and special specimens at OOALC.

Statistical analysis of the test data showed that the physical properties of the new source conpared closely to the old source of UF-2121 liner specimens.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 Rev A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.